

FORM PTO-1390 (REV. 11-2000)	U S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		Mo-5487/WW-5382 U S APPLICATION NO (if known, see 37 CFR 1.5 <b>To be Assigned</b> <b>09/914436</b>
INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/EP00/01481	23 February 2000 (23.02.00)	03 March 1999 (3.03.99)
TITLE OF INVENTION METHOD FOR PRODUCING COMPACTED FREE-FLOWING RAW MATERIALS FOR VARNISH		
APPLICANT(S) FOR DO/EO/US 1) Lutz Hoppe; 2) Martin Lohrie; 3) Lutz Riechadt; 4) Holger Tanneberger		
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:		
<p>1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</p> <p>2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.</p> <p>3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.</p> <p>4. <input checked="" type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31).</p> <p>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2))           <ul style="list-style-type: none"> <li>a. <input checked="" type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau).</li> <li>b. <input type="checkbox"/> has been communicated by the International Bureau.</li> <li>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</li> </ul> </p> <p>6. <input checked="" type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).           <ul style="list-style-type: none"> <li>a. <input checked="" type="checkbox"/> is attached hereto.</li> <li>b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4).</li> </ul> </p> <p>7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))           <ul style="list-style-type: none"> <li>a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau).</li> <li>b. <input type="checkbox"/> have been communicated by the International Bureau.</li> <li>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</li> <li>d. <input type="checkbox"/> have not been made and will not be made.</li> </ul> </p> <p>8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).</p> <p>9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).</p> <p>10. <input type="checkbox"/> An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).</p>		
<b>Items 11 to 20 below concern document(s) or information included:</b>		
<p>11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</p> <p>12. <input checked="" type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</p> <p>13. <input checked="" type="checkbox"/> A FIRST preliminary amendment.</p> <p>14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.</p> <p>15. <input type="checkbox"/> A substitute specification.</p> <p>16. <input type="checkbox"/> A change of power of attorney and/or address letter.</p> <p>17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.</p> <p>18. <input checked="" type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4).</p> <p>19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).</p> <p>20. <input checked="" type="checkbox"/> Other items or information:</p>		
PTO Form 1449 and references listed therein; Preliminary Amendment w/Abstract; 1 page of drawings containing Figure 1		

U.S. APPLICATION NO. (if known, see 37 CFR 1.57)  
To Be Assigned 0014436INTERNATIONAL APPLICATION NO  
PCT/EP00/01481ATTORNEY'S DOCKET NUMBER  
Mo-5487/WW-538221.  The following fees are submitted:**BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):**

Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... \$1000.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... \$860.00

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$710.00

International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$690.00

International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00

**ENTER APPROPRIATE BASIC FEE AMOUNT =****CALCULATIONS PTO USE ONLY**

Surcharge of \$130.00 for furnishing the oath or declaration later than  20  30 months from the earliest claimed priority date (37 CFR 1.492(e)).

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$
Total claims	9 -20 =	0	x \$18.00	\$ 0.00
Independent claims	1 -3 =	0	x \$80.00	\$ 0.00
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$270.00	\$ 0.00

**TOTAL OF ABOVE CALCULATIONS =**

<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.	+	\$ 0.00
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**SUBTOTAL =**

Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).	\$ 860.00
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**TOTAL NATIONAL FEE =**

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property	+	\$ 40.00
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**TOTAL FEES ENCLOSED =**

	Amount to be refunded:	\$
	charged:	\$

- a.  A check in the amount of \$ \_\_\_\_\_ to cover the above fees is enclosed.
- b.  Please charge my Deposit Account No. 13-3848 in the amount of \$ 900.00 to cover the above fees. A duplicate copy of this sheet is enclosed.
- c.  The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 13-3848. A duplicate copy of this sheet is enclosed.
- d.  Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

**NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.**

SEND ALL CORRESPONDENCE TO

SIGNATURE

Aron Preis

NAME

29,426

REGISTRATION NUMBER



00157

PATENT TRADEMARK OFFICE

09/914436

JOG3 REC'D 27 AUG 2001 27 AUG 2001

PATENT APPLICATION  
Mo-5487  
WW-5532

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

APPLICATION OF )  
LUTZ HOPPE ET AL ) PCT/EP00/01481  
SERIAL NUMBER: TO BE ASSIGNED )  
FILED: HEREWITH )  
TITLE: METHOD FOR PRODUCING )  
COMPACTED FREE-FLOWING )  
RAW MATERIALS FOR VARNISH )  
)

**PRELIMINARY AMENDMENT**

Assistant Commissioner for Patents

Washington, D.C. 20231

Sir:

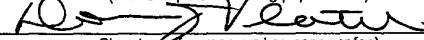
This preliminary amendment is being filed concurrently with the subject patent application. Upon granting a Serial Number and filing date, please amend the subject patent application as follows.

"Express Mail" mailing label number ET14689843US  
Date of Deposit August 27, 2001

I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Assistant Commissioner of Patents and Trademarks, Washington, D.C. 20231

Lorraine J. Veatch

(Name of person mailing paper or fee)



Signature of person mailing paper or fee)

Please amend the Application as follows.

**IN THE ABSTRACT:**

Please replace the abstract with the following.

**--METHOD FOR PRODUCING COMPACTED FREE-FLOWING  
RAW MATERIALS FOR VARNISH**

**ABSTRACT OF THE DISCLOSURE**

A process for producing compacted free-flowing nitrocellulose based lacquer raw materials is described. The process involves pressing a nitrocellulose based lacquer feed material, which is moistened with alcohol or water, through the holes of a die. The pressing step may be performed by means of at least one circulating breaker. The process optionally comprises shearing off the compacted lacquer raw material into pieces of selected length, below the die. The compacted nitrocellulose based lacquer raw materials prepared by the process of the present invention are free flowing and have a moisture content of at least 25 %.--

A separate abstract page is included herewith.

**IN THE SPECIFICATION:**

Please replace the title at line 1, page 1 of the specification with the following.

**--METHOD FOR PRODUCING COMPACTED FREE-FLOWING RAW MATERIALS  
FOR VARNISH--**

Please insert the following between lines 1 and 3 on page 1 of the specification.

**--CROSS REFERENCE TO RELATED PATENT APPLICATIONS**

The present patent application claims the right of priority under 35 U.S.C. 119 and 35 U.S.C. 365 of International Application No. PCT/EP00/01481, filed 23 February 2000, which was published in German as International Patent Publication No. WO 00/52058 on 8 September 2000, which is entitled to the right of priority of German Patent Application No. 199 09 230.3, filed 3 March 1999.

## **FIELD OF THE INVENTION--**

Please insert the following at line 6 on page 1 of the specification.

## **--BACKGROUND OF THE INVENTION--**

Please insert the following at line 26 on page 2 of the specification.

## **--SUMMARY OF THE INVENTION--**

Please replace the paragraph at lines 1-8 on page 3 of the specification with the following.

--It has now surprisingly been found that compacted nitrocellulose can also be obtained by causing the circulating breakers (or oscillating wheels) in a breaker mill, which breakers travel on a die (plate) which is provided with holes, to press the moistened nitrocellulose through the holes (e.g. bores) in the die (see Figure 1). The nitrocellulose lacquer raw material is thereby compacted. Underneath the die there is a shearing-off apparatus by means of which the granule-like preforms are brought to the desired length. The cross-sectional shape of the preforms is determined by the shape of the hole cross-section.--

Please insert the following between lines 8 and 10 on page 3 of the specification.

## **--BRIEF DESCRIPTION OF THE DRAWING**

Fig. 1 is a schematic representation of a breaker mill that may be used in the process of the present invention. The reference characters of Figure 1 are summarized as follows: (1) represents the drive shaft; (2) represents a breaker; (3) represents the die with holes; (4) represents the shearing-off apparatus; (5) represents the housing; d represents the bore diameter; l represents the bore length;  $D_K$  represents the breaker diameter; B represents the breaker width; and  $D_M$  represents the die diameter.

## **DETAILED DESCRIPTION OF THE INVENTION--**

Please delete lines 13-24 on page 4 of the specification.

Please replace line 1 on page 7 of the specification with the following.

--WHAT IS CLAIMED IS--

**IN THE CLAIMS:**

Please add the following Claims 8 and 9.

--8. The process of Claim 2 wherein said pressing ratio P is from 0.5 to 3.0.

9. The process of Claim 1 wherein the compacted free-flowing lacquer raw material has a moisture content of at least 25 %.--

Please replace Claim 1 with the following.

1. (Once Amended, Clean) A process for producing compacted free-flowing lacquer raw materials based on nitrocellulose comprising pressing lacquer raw material, which is moistened with alcohol or water, through the holes of a die.

Please replace Claim 2 with the following.

2. (Once Amended, Clean) The process of Claim 1 wherein said process is carried out at a pressing ratio, P, as represented by the following formula,

$$P = (\text{length of bore}) \div (\text{diameter of bore})$$

of from 0.5 – 5.0.

Please replace Claim 3 with the following.

3. (Once Amended, Clean) The process of Claim 1 wherein the lacquer raw material is pressed through the die holes by means of one or more circulating breakers.

Please replace Claim 4 with the following.

4. (Once Amended, Clean) The process of Claim 1 wherein the lacquer raw material is pressed through the die holes by means of one or more oscillating wheels.

Please replace Claim 5 with the following.

5. (Once Amended, Clean) The process of Claim 1 further comprising shearing off the compacted lacquer raw material, below the die, into pieces of selected length.

Please replace Claim 6 with the following.

6. (Once Amended, Clean) The process of Claim 1 wherein the lacquer raw material has a nitrogen content  $\leq$  12.6 %.

Please replace Claim 7 with the following.

7. (Once Amended, Clean) A compacted free-flowing lacquer raw material based on nitrocellulose prepared by the process of Claim 1.

## REMARKS

Claims in the case are 1-9, upon entry of the present amendment. Claims 8 and 9 have been added, and Claims 1-7 have been amended herein.

Claims 1-7 of the above-identified patent application have been amended as to form, for example, by introducing indefinite and definite articles, replacing "characterized in that" with --wherein--, and converting multi-dependent claims to singly dependent claims. Basis for added Claim 8 is found in original Claim 2, and at page 3, lines 24-30 of the specification. Basis for added Claim 9 is found at page 3, lines 10-12 of the specification.

Page 1 of the application has been amended herein to introduce cross reference information. The cross reference information is presented in accordance with 37 C.F.R. 1.78(a)(2) (Federal Register / Vol. 65, No. 183 / Wednesday, September 20, 2000; Changes to Implement Eighteen-Month Publication of Patent Applications; Final Rule).

The title of the application has been changed to correspond with that of the related International Patent Publication No. WO 00/52058. The specification has been amended to include section headings. A brief description of the drawing has also been included in the specification, basis for which is found in Figure 1, and at page 4, lines 13-24 of the original specification. The heading of the claims section of the specification has been changed from "Claims" to --WHAT IS CLAIMED IS:--. Basis for the addition of --or oscillating-- prior to "wheels" at line 2, page 3 of the specification is found in original Claim 4. The abstract of the patent application has been replaced, and the new abstract is included herewith on a separate page.

The amendments presented herein are not believed to represent the entry of new matter into the application. Applicants respectfully request entry of this Preliminary Amendment.

Respectfully submitted,

LUTZ HOPPE  
MARTIN LOHRIE  
LUTZ RIECHARDT  
HOLGER TANNEBERGER

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/jme/JRF0084

## **VERSIONS WITH MARKINGS TO SHOW CHANGES MADE**

### **IN THE SPECIFICATION:** (Marked-Up)

The following are additions and changes that have been made to the specification.

The title at line 1, page 1 of the specification has been amended as follows.  
[A process for producing compacted, free-flowing lacquer raw materials]

### **METHOD FOR PRODUCING COMPACTED FREE-FLOWING RAW MATERIALS FOR VARNISH**

The following has been inserted between lines 1 and 3 on page 1 of the specification.

### **CROSS REFERENCE TO RELATED PATENT APPLICATIONS**

The present patent application claims the right of priority under 35 U.S.C. 119 and 35 U.S.C. 365 of International Application No. PCT/EP00/01481, filed 23 February 2000, which was published in German as International Patent Publication No. WO 00/52058 on 8 September 2000, which is entitled to the right of priority of German Patent Application No. 199 09 230.3, filed 3 March 1999.

### **FIELD OF THE INVENTION**

The following has been inserted at line 6 on page 1 of the specification.

### **BACKGROUND OF THE INVENTION**

The following has been inserted at line 26 on page 2 of the specification.

### **SUMMARY OF THE INVENTION**

The paragraph at lines 1-8 on page 3 of the specification has been amended as follows.

It has now surprisingly been found that compacted nitrocellulose can also be obtained by causing the circulating breakers (or oscillating wheels) in a breaker mill,

which breakers travel on a die (plate) which is provided with holes, to press the moistened nitrocellulose through the holes (e.g. bores) in the die (see Figure 1). The nitrocellulose lacquer raw material is thereby compacted. Underneath the die there is a shearing-off apparatus by means of which the granule-like preforms are brought to the desired length. The cross-sectional shape of the preforms is determined by the shape of the hole cross-section.

The following has been inserted between lines 8 and 10 on page 3 of the specification.

#### BRIEF DESCRIPTION OF THE DRAWING

Fig. 1 is a schematic representation of a breaker mill that may be used in the process of the present invention. The reference characters of Figure 1 are summarized as follows: (1) represents the drive shaft; (2) represents a breaker; (3) represents the die with holes; (4) represents the shearing-off apparatus; (5) represents the housing; d represents the bore diameter; l represents the bore length; D<sub>K</sub> represents the breaker diameter; B represents the breaker width; and D<sub>M</sub> represents the die diameter.

#### DETAILED DESCRIPTION OF THE INVENTION

The following lines 13-24 on page 4 of the specification have been deleted.

[Figure 1:

- (1): drive shaft
  - (2): breaker
  - (3): die with holes
  - (4): shearing-off apparatus
  - (5): housing
- d: bore diameter  
e: bore length  
D<sub>K</sub>: breaker diameter

Line 1 on page 7 of the specification has been amended as follows.

[Claims] WHAT IS CLAIMED IS:

**IN THE CLAIMS:** (Marked-Up)

The following are versions of the amended claims with markings to show changes made thereto in the present Preliminary Amendment.

1. (Once Amended, Marked-Up) A process for producing compacted free-flowing lacquer raw materials based on nitrocellulose[, characterised in that] comprising pressing [the] lacquer raw material, which is moistened with alcohol or water, [is pressed] through the holes of a die [provided with holes].

2. (Once Amended, Marked-Up) [A] The process [according to claim] of Claim 1[, characterised in that the] wherein said process is carried out at a pressing ratio, P, as represented by the following formula,

$$P = \frac{\text{[length of bore}}{\text{diameter of bore}} =$$

$$P = (\text{length of bore}) \div (\text{diameter of bore})$$

of from 0.5 – 5.0[, and preferably ranges between 0.5 and 3.0].

3. (Once Amended, Marked-Up) [A] The process [according to either one] of [claims 1 or 2, characterised in that] Claim 1 wherein the lacquer raw material is pressed through the die holes by means of one or more circulating breakers.

4. (Once Amended, Marked-Up) [A] The process [according to either one] of [claims 1 or 2, characterised in that] Claim 1 wherein the lacquer raw material is pressed through the die holes by means of one or more oscillating wheels.

5. (Once Amended, Marked-Up) [A] The process [according to any one] of [claims 1 to 4, characterised in that] Claim 1 further comprising [the compacted lacquer raw material is sheared] shearing off the compacted lacquer raw material, below the die, into pieces of [the desired] selected length.

5. (Once Amended, Marked-Up) [A] The process [according to any one] of [claims 1 to 4, characterised in that] Claim 1 further comprising [the compacted lacquer raw material is sheared] shearing off the compacted lacquer raw material, below the die, into pieces of [the desired] selected length.

6. (Once Amended, Marked-Up) [A] The process of Claim 1 wherein [for producing] the lacquer raw material[s] in the form of granules, characterised in that the nitrocellulose, which is moistened with water or alcohol and which is used as the lacquer raw material,] has a nitrogen content  $\leq$  12.6 %.

7. (Once Amended, Marked-Up) A compacted[,] free-flowing lacquer raw material based on nitrocellulose[, obtainable] prepared by [a] the process [according to any one of claims 1 to 6] of Claim 1.

8. (Added) The process of Claim 2 wherein said pressing ratio P is from 0.5 to 3.0.

9. (Added) The process of Claim 1 wherein the compacted free-flowing lacquer raw material has a moisture content of at least 25 %.

METHOD FOR PRODUCING COMPACTED FREE-FLOWING  
RAW MATERIALS FOR VARNISH

ABSTRACT OF THE DISCLOSURE

A process for producing compacted free-flowing nitrocellulose based lacquer raw materials is described. The process involves pressing a nitrocellulose based lacquer feed material, which is moistened with alcohol or water, through the holes of a die. The pressing step may be performed by means of at least one circulating breaker. The process optionally comprises shearing off the compacted lacquer raw material into pieces of selected length, below the die. The compacted nitrocellulose based lacquer raw materials prepared by the process of the present invention are free flowing and have a moisture content of at least 25 %.

1/PAT

- 1 -

09/914436  
JC03 Reg'd PCT TTO 27 AUG 2001A process for producing compacted, free-flowing lacquer raw materials

The present invention relates to a process for producing compacted free-flowing nitrocellulose, which is moistened with water or alcohol, by pressing it through a plate provided with holes.

5

Nitrocellulose materials which have a low degree of esterification and a nitrogen content of up to 12.6 %, and which are mainly used in the lacquer industry, are produced by the esterification of cellulose materials with nitrating acids, which usually consist of a mixture of nitric acid, sulphuric acid and water.

10

After the nitrating acid has been removed, generally by intensive washing with water, and after the desired molecular weight of the nitrocellulose has been obtained by a thermal decomposition process, the nitrocellulose which is thus obtained, and which has a fibrous structure, has to be stabilised to prevent self-ignition. Various stabilising agents are used for this purpose.

15

20

Apart from the incorporation of plasticisers by admixture, the most commonly used method is the moistening of the nitrocellulose with alcohols and/or water. The nitrocellulose materials are usually sold with a moisture content of alcohols (such as ethanol, isopropanol or butanol for example) and/or water of 30 or 35 %, respectively. If the degree of moistening is less than 25 %, these partially esterified nitrocellulose materials have to be treated as "explosive substances" due to their increased hazard potential (Recommendations on the Transport of Dangerous Goods, 10th Edition, United Nations (1997)).

25

30

Due to their wool-like structure, fibrous nitrocellulose materials have apparent densities which range between 250 and 350 g/l on average. With regard to the shipping of products such as these, their low apparent density has a disadvantageous effect on packing and shipping costs. This is countered by tamping the fibrous nitrocellulose into the packaging container, such as a drum or carton for example.

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Date of Deposit August 27, 2001

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Donna J. Veatch

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Donna J. Veatch

Although the apparent density is in fact increased in this manner, the pourability of the nitrocellulose is at the same time reduced. This results in an increased labour requirement for emptying the nitrocellulose container.

- 5 A process for treating fibrous nitrocellulose materials is known, the object of which is to make them safer for transport and storage (GB-B-871 299). Compaction is effected by exerting a compressive force  $P = 2M + 6400$  on the moist, fibrous nitrocellulose, where P is the force in pounds per square inch and M is the mean fibre length of the nitrocellulose in microns. The force, which preferably ranges between  
10 15,000 and 17,000 psi ( $1110-1196 \text{ Kp/cm}^2$ ), is applied by two rolls which are driven in counter-rotation without a separation.

- 15 The flake-like nitrocellulose which is thus obtained subsequently has to be broken into smaller pieces in a processing unit. Apart from its high capital and operating costs, one considerable disadvantage of this process is the drying out of the nitrocellulose which occurs during roll compaction. In GB PS 871 299, all the compacted nitrocellulose materials which are obtained according to the examples have a moisture content less than 25 %, and are consequently explosive substances. It has been shown in practice that roll compaction under the action of high pressures results in self-ignition phenomena which constitute a hazard for personnel and for the plant, particularly since there is always a very large amount of nitrocellulose in the roll gap.  
20  
25

- A similar process principle is described in US PS 5 378 826. The aforementioned disadvantages are also applicable there.

The object of the present invention was therefore to provide a new process for producing compacted, free-flowing nitrocellulose which prevents the nitrocellulose from drying out.

It has now surprisingly been found that compacted nitrocellulose can also be obtained by causing the circulating breakers (wheels) in a breaker mill, which breakers travel on a die (plate) which is provided with holes, to press the moistened nitrocellulose through the holes (e.g. bores) in the die (see Figure 1). The  
5      nitrocellulose lacquer raw material is thereby compacted. Underneath the die there is a shearing-off apparatus by means of which the granule-like preforms are brought to the desired length. The cross-sectional shape of the preforms is determined by the shape of the hole cross-section.

10      It should be emphasised that if defined process parameters are maintained there is hardly any drying-out of the nitrocellulose which is used, and thus the latter does not fall within the explosive substance range of less than 25 % moisture.

15      At least one breaker rotates in the breaker mill. Two breakers are customary, although more than 2 breakers can be provided. This depends on the size of the processing unit and on the diameter of the breakers. The compacted material which is obtained can vary from hard (with sharp edges) to soft (easily crushed with the fingers).

20      It is also possible, however, for the moistened nitrocellulose simply to be pressed through the die and thus to be compacted by a reciprocating movement of a wheel on a die which is provided with holes.

25      When the die contains circular holes, the pressing ratio P determines the consistency of the compacted nitrocellulose. P is defined as the ratio of the length of the bore to the diameter of the bore in the die:

$$P = \frac{\text{length of bore}}{\text{diameter of bore}} .$$

30      The pressing ratio P should range between 0.5 – 5.0, preferably between 0.5 and 3.0.

It is also possible for the holes in the die to have a square, rectangular, oval or irregularly shaped cross-section. The number of holes per unit area of the die depends  
5 on the stability of the die. By means of compaction, it is possible to increase the apparent density of the nitrocellulose used by a multiple and thereby to obtain free-flowing material.

The uncompacted material is fed into the breaker mill via a metering device (such as a continuous screw or belt for example). It is also possible to provide the breaker mill  
10 with an inert atmosphere, e.g. nitrogen or carbon dioxide. The following examples describe the process in greater detail, but do not limit it.

**Figure 1:**

- 15 (1): drive shaft  
(2): breaker  
(3): die with holes  
(4): shearing-off apparatus  
(5): housing

20 d: bore diameter

e: bore length

D<sub>K</sub>: breaker diameter

B: breaker width

D<sub>M</sub>: die diameter

**Example 1**

Nitrocellulose corresponding to Standard A 30, moistened with 34.6 % ethanol, was continuously fed at 210 kg/h into the breaker mill (die diameter:  $D_M = 175$  mm, breaker diameter:  $D_K = 130$  mm, breaker width:  $B = 27$  mm, number of breakers: 2, drive shaft speed = 150 rpm). The pressing ratio was 2 (hole diameter:  $d = 6$  mm, hole shape: circular, length of bore:  $l = 12$  mm). The distance from the shearing apparatus to the underside of the die was 20 mm. The compacted material had a moisture content of 34.2 % and an apparent density of 578 g/l. The preforms remained free-flowing in a 100 litre drum even after being stored therein for eight weeks.

**Examples 2 – 7**

These examples were carried out as described for Example 1. The test parameters are given in Table 1.

Example	NC Standard	Moistening agent	Moisture content		Throughput	Apparent density		Die	Drive shaft speed (rpm)		
			before (%)	after (%)	moist NC (kg/hour)	before (g/l)	after (g/l)	D (mm)	L (mm)	P	
1	A 30	ethanol	34.6	34.2	210	383	578	6	12	2	150
2	E 27	ethanol	32.4	31.5	116	253	565	6	12	2	150
3	E 22	ethanol	32.0	31.7	130	196	556	10	20	2	150
4	E 34	ethanol	34.1	31.9	332	438	602	10	20	2	150
5	E 24	isopropanol	35.2	35.0	190	191	556	8	16	2	150
6	A 27	ethanol	30.9	30.4	133	380	539	6	6	1	150
7	E 22	ethanol	32.8	31.7	57	196	526	6	6	1	201

**Claims**

- 5        1. A process for producing compacted free-flowing lacquer raw materials based on nitrocellulose, characterised in that the lacquer raw material, which is moistened with alcohol or water, is pressed through a die provided with holes.

- 10      2. A process according to claim 1, characterised in that the pressing ratio P

$$P = \frac{\text{length of bore}}{\text{diameter of bore}} = 0.5 - 5.0, \text{ and preferably ranges between } 0.5 \\ \text{and } 3.0.$$

- 15      3. A process according to either one of claims 1 or 2, characterised in that the lacquer raw material is pressed through the die holes by means of one or more circulating breakers.

- 20      4. A process according to either one of claims 1 or 2, characterised in that the lacquer raw material is pressed through the die holes by means of one or more oscillating wheels.

- 25      5. A process according to any one of claims 1 to 4, characterised in that the compacted lacquer raw material is sheared off below the die into pieces of the desired length.

- 25      6. A process for producing lacquer raw materials in the form of granules, characterised in that the nitrocellulose, which is moistened with water or alcohol and which is used as the lacquer raw material, has a nitrogen content  $\leq 12.6\%$ .

- 30      7. A compacted, free-flowing lacquer raw material based on nitrocellulose, obtainable by a process according to any one of claims 1 to 6.

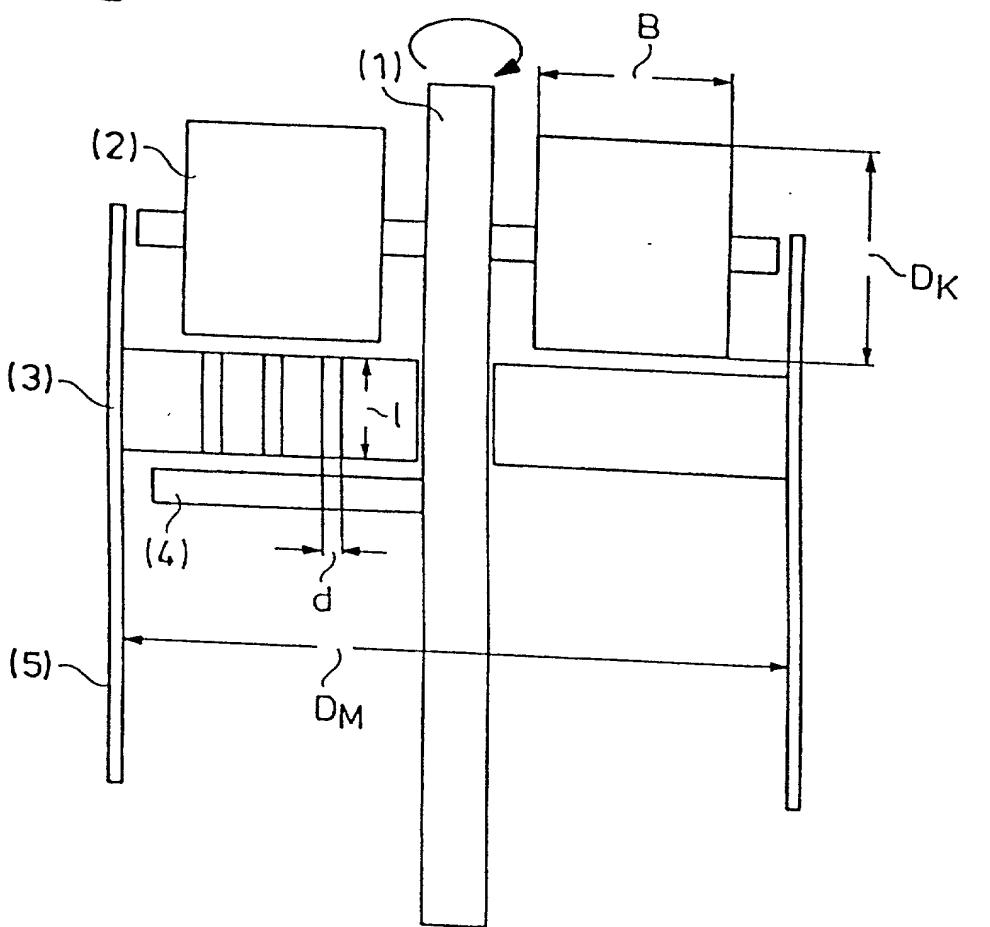
**A process for producing compacted free-flowing lacquer raw materials**

**Abstract**

A process for producing compacted free-flowing lacquer raw materials, characterised in that the lacquer raw material, which is moistened with water or alcohol, is pressed through a die provided with holes.

09/914436

Fig. 1



**POWER OF ATTORNEY:** As a named inventor, I hereby appoint the following attorney(s) and this application and to transact all business in the Patent and Trademark Office conne

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**COMBINED DECLARATION AND POWER OF ATTORNEY**

ATTORNEY DOCKET NO

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought

on the invention entitled

**METHOD FOR PRODUCING COMPACTED FREE-FLOWING RAW MATERIALS FOR VARNISH**

the specification of which is attached hereto,

or was filed on **February 23, 2000**

as a PCT Application Serial No. **PCT/EP00/01481**

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

<b>199 09 230.3</b> (Number)	<b>Germany</b> (Country)	<b>March 3, 1999</b> (Month/Day/Year Filed)
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I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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